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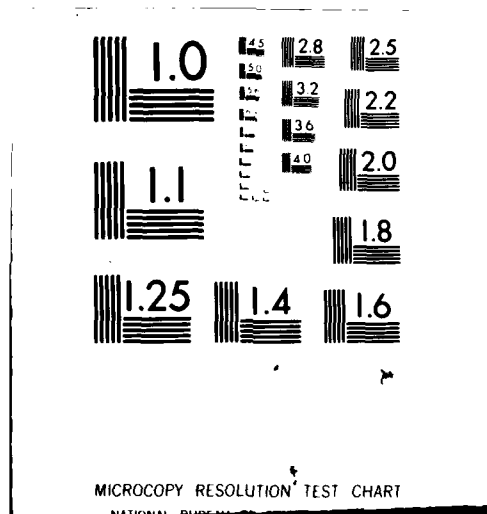UNCLASSIFIED   MRL-R-783   NL

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MELBOURNE, VICTORIA

REPORT

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HYDROCRACKING OF THE OILS OF BOTRYOCOCCUS BRAUNII

TO TRANSPORT FUELS

L.W. Hillen, G. Pollard, L.V. Wake and N. White

Approved for Public Release

JULY, 1980

DEPARTMENT OF DEFENCE

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16. ABSTRACT *(if this is security classified, the announcement of this report will be similarly classified):*

Hydrocarbon oils of the alga *Botryococcus braunii*, extracted from a natural 'bloom' of the plant, have been hydrocracked to produce a distillate comprising 67% a petrol fraction, 15% an aviation turbine fuel fraction, 15% a diesel fuel fraction and 3% residual oil. The distillate was examined by a number of standard petroleum industry test methods. This preliminary investigation indicates that the oils of *B. braunii* are suitable as a feedstock material for hydrocracking to transport fuels.

C O N T E N T S

* * *

HYDROCRACKING OF THE OILS OF BOTRYOCOCCUS

BRAUNII TO TRANSPORT FUELS

1. INTRODUCTION

The phytoplanktonic species *Botryococcus braunii* is a prolific hydro-carbon-producing plant which has been suggested as a potential energy crop [1]. The hydrocarbon oils of *B. braunii* are high boiling liquids which have been loosely described as equivalent to the 'gas-oil fraction of crude oil'.

Before a detailed biological research programme can be undertaken, the question arises concerning the suitability of the algal oil as a renewable source of transport fuels. Therefore as part of the preliminary assessment of *B. braunii*, a sample of the plant's oil has been subjected to a hydro-cracking procedure and the product fractions tested as to their suitability for use as petrol and aviation turbine fuels.

2. MATERIALS AND METHODS

2.1 Algal Material

The *B. braunii* oils used for the present study were obtained from plant material harvested from the Darwin River Reservoir, Northern Territory during October 1977. The characteristics of the algal bloom which occurred on this reservoir over the period 1975-79 have been described elsewhere [2]. After collection, the algal colonies were freeze-dried. The hydrocarbons, which constituted 30% of the algal mass, were extracted with acetone and purified by column chromatography on alumina by the method of Brown *et al.* [3].

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2.2 Hydrocracking Treatment of the Oil

The apparatus, which was used for the hydrocracking experiment, has been previously employed for the hydrogenation of coal and coal derived liquids. It has been described in detail elsewhere [4]. A simplified flow diagram is shown in Figure 1. In short, the reactor was a 6.5 m coiled stainless steel tube of 6.35 mm diameter, packed with 120 g of a cobalt molybdate catalyst (HT/400E, 1/16", Harshaw Chemical Co.). The oil and hydrogen gas at 20 MPa (3000 psi) were passed through the reactor which was held at 400°C. The liquid products were cooled and collected in knock-out pots, surplus hydrogen being vented. A total of 160 g of oil sample was fed to the hydrocracker at the rate of 230 ml/hour and 127 g of product was obtained. Variations in column temperature were noted and are shown in Table 1.

Both the substrate, i.e. the uncracked algal oil and the hydrocracked products were subjected to a 'true boiling point distribution' determination by gas chromatography (G.C.) according to the method of Jackson et al. [5]. These workers had earlier found excellent agreement between boiling point distribution by G.C. with true boiling point curves obtained by conventional distillation.

The liquid hydrocarbons were also examined by the American Society for Testing Materials (ASTM) and the American Petroleum Institute (API) test methods for product suitability as set out in Table 2. In addition the products were analysed by gas chromatography-mass spectrometry (GC-MS) employing a Finnigan 3300-E with a 6100 information collection system equipped with chemical ionization using methane gas. Each spectrum was compared with standard mass spectra [6] with the aim of assigning structures where possible.

3. RESULTS AND DISCUSSION

Analyses of the *B. braunii* oil as extracted from the plant are provided in Table 3. The structural characteristics of the oil's components have not yet been fully evaluated but mass spectral data suggests they are all closely related to botryococcene, Table 3 and Fig. 2 (see also 1). Elemental analysis of the oil revealed that the nitrogen and sulphur values were at levels which are considered most unlikely to affect operation of the catalyst (see Table 3).

Figure 3 compares the boiling point (BP) variation of the cracked oil with that of the uncracked feed-oil as well as the BP variation of a typical Bass Strait crude-oil. The figure indicates that, in the actual sample of plant oil to be hydrocracked, not all of the low boiling hydrocarbon solvent (hexane) used during the column chromatographic purification of the oil was removed by the rotary evaporator. The presence of this low boiling solvent in the feed-oil (Table 4), while disconcerting, does not alter the general conclusions to be drawn from the experiment.

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Upon charging, the reactor cooled slightly which is considered to be related to partial volatilisation of the uncracked oil (Table 1). A temperature rise was subsequently observed one metre down the column, presumably as a consequence of the hydrogenation of the alkene components, this being an exothermic process [7]

$$e.g. \quad CH_2 = CH_2 \quad + \quad H_2 \quad \rightarrow \quad CH_3 - CH_3 \quad\quad -30 \text{ KCAL}$$

The observed temperature rise along the column suggests that saturation of the hydrocarbon took place prior to cracking. The catalyst used is known primarily as a hydrogenation agent with only mild cracking properties. The minor temperature rise half way down the column is believed to be associated with ring formation and/or hydrogenation of the cracked products. The degree of unsaturation would be lower at this stage than in the original feed oil.

The properties of the liquid hydrocarbons produced by processes such as hydrogenation vary, depending on the temperature and hydrogen pressure, as well as the type of catalyst employed. As can be seen from Table 5, the transport fuels gasoline, kerosine and diesel distillate comprised 97% of the product. In this first run, the conditions employed resulted in the production of two-thirds of the product volume as gasoline and approximately one sixth each as kerosine and diesel distillate. A very low concentration of aromatics was present in the hydrocracked material unlike similarly treated materials obtained from coal sources (e.g. Table 11).

The hydrogen pressures used and temperature of cracking were probably more severe than would actually be needed in practice as evidenced by the high petrol fraction yield in the product boiling point distribution (Table 6, Figure 3). As the catalyst reportedly had only mild cracking properties it is evident that the hydrocarbons of *B. braunii* readily undergo cleavage. This is in keeping with the known cracking properties of isoparaffins, of which botryococcene is a highly branched member, compared to normal paraffins [8] and indicates that the plant would make a suitable feedstock. Due to the small sample size and lack of ageing of the catalyst, the results obtained must be treated as illustrative rather than definitive for the cracking characteristics of the feed oil.

Analytical data on the hydrocracked oils are provided in Tables 5 to 10. Table 9 lists individual hydrocarbon compounds identified from GC retention index data. Those that have been confirmed by GC-MS are marked with an asterisk. Interpretation of the observed product spread of Table 9 must await more detailed information on the chemical structure of the feed-oil components.

As insufficient material was available for the determination of the octane rating of the petrol fraction by an engine test method, the value was estimated by regression analysis (9 and 10) utilising the data from Table 9. The composition and octane rating of the gasoline fraction are compared in Table 11 with gasolines derived from the hydrogenation of brown and black coal tars and also with the straight run gasoline fraction of a Bass Strait crude oil [4]. In preparing Table 11, the components which were shown as

3

'not identified' in Tables 9 and 10 have been apportioned to paraffins and naphthenes in the same ratio as the identified fractions. GC-MS studies indicated that the unidentified components were a mixture of C_nH_{2n} and C_nH_{2n+2} hydrocarbons. As FIA analysis had previously shown the olefins to be insignificant (Table 6), the C_nH_{2n} compounds can be taken to be naphthenes. In apportioning the 'unidentifieds' in this manner, the GC-MS results suggest that the paraffin content, as shown in Table 11, can be assumed to be a maximum figure and the naphthene figure to be a minimum.

Table 11 draws attention to the fact that even gasoline fractions derived from a mineral oil require further processing in order to meet product specifications (in this case octane number). Thus the need to further refine *Botryococcus* oils to yield specification transport fuels does not necessarily detract from the plant's potential as an energy crop.

From Table 8 the most interesting aspect of the product is the low aromatic content and the virtual absence of olefins. The higher octane rating for this material compared to straight run Bass Strait crude reflects the lower n-hydrocarbon content (<8%, Table 9) of the treated algal oil. Normal hydrocarbons account for 70% of the paraffins of Bass Straight crude [8]. It would therefore appear that petrols derived from *B. braunii* will be useful as blendstock additives.

The low yield of the aviation turbine fuel fraction allowed only a few simple tests to be carried out. The cut easily meets the density and the aromatic content requirements for turbine fuel. Given the wide range of components in the petrol fraction (Table 9), the freezing point of the 'AVTUR' fraction is probably close to specification. However it is the low aromatic figure which is of prime importance, emphasizing as it does the potential for hydrocracked *B. braunii* oil to be used as a blendstock in the production of aviation fuels where specifications call for a figure of less than 20% aromatics (DEF AUST 208 B).

4. CONCLUSIONS

The oils of *B. braunii* are suitable as a feed oil for use in a hydro-cracker. The products are low in aromatic hydrocarbons and would appear to be potentially suitable for use as a blendstock especially in the production of aviation turbine fuels. A larger scale evaluation would be needed to adequately define the cracked product type and to evaluate catalyst degradation.

No serious technical problems associated with the raw plant oil are envisaged at this stage which would mitigate against *B. braunii* being used as a potential energy crop. From this preliminary study, the algal oils appear suitable as hydrocarbon feedstock materials for conversion to specification transport fuels.

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    and 2b2.1).

T A B L E   1

HYDROCARBONS FROM ALGAL BLOOMS OF B. BRAUNII

Retention Index	Compound	Figure No.	Molecular Mass	Percentage Composition			
				Tarago	Sorrento Green 76	Sorrento Red 76	Sorrento 78 Sample
2467	$C_{25}H_{48}$	1	348	2	2	1	7
2666	$C_{27}H_{50}$	–	374	3	–	–	–
2673	$C_{27}H_{52}$	2	376	40	18	–	56
2858	$C_{29}H_{54}$	3	402	13	37	27	23.5
2874	$C_{29}H_{56}$	4	404	37	"	"	6.5
3046	$C_{31}H_{58}$	5	430	–	16	8	4
3071	$C_{31}H_{60}$	6	432	5	"	"	3
3237[+]	–	–	–	–	27	64	–
TOTALS				100	100	100	100
Percentage Oil Content				30+3	27	NE	30

NE   Not examined.

+   Not positively identified by MS.

"   Inverted commas indicate peak not resolved from immediately preceding figure.

T A B L E   2

ASTM AND API ANALYSIS

Description	Analysis Reference	Sample Fraction	Table No
Distillation	ASTM-D86-67	$< 191^{\circ}C$	Table 6
	modified by using 10 ml and 20 ml samples	$191-232^{\circ}C$	Table 8
Density, S.G. or API gravity by hydrometer	ASTM-D1298-67	Total sample	Table 5
Density and S.G. by Lipkin	ASTM-D941-55	$< 191^{\circ}C$	Table 6
bicapillary pycnometer		$191-232^{\circ}C$	Table 8
Tests for hydrocarbon	ASTM-D1319-70	$< 191^{\circ}C$	Table 6
types by FIA		$191-232^{\circ}C$	Table 8
Volumetric average[+] boiling point,	API Ref 9	$< 191^{\circ}C$	Table 7
Mean average boiling point[+]	API Ref 9	$< 191^{\circ}C$	Table 7
C:H mass ratio,	API Ref 9	$< 191^{\circ}C$	Table 7
Aniline point,*	API Ref 9	$< 191^{\circ}C$	Table 7
Molecular weight,	API Ref 9	$< 191^{\circ}C$	Table 7
Aniline gravity constant[++]	API Ref 9	$< 191^{\circ}C$	Table 7
Watson K factor**	API Ref 9	$< 191^{\circ}C$	Table 7
True boiling point distribution[#]	API Ref 5	Total sample	Table 5

T A B L E   2

(Continued)

+ The average boiling point distillations are used to define the volatility characteristics of petroleum fractions. These are batch distillations which differ from ASTM distillations mainly in the degree of fractionation obtained.

* The aniline point is the lowest temperature at which the fraction is miscible with an equal volume of aniline. It is a measure of paraffinicity.

++ The aniline gravity constant is an estimation of combustion based on an empirical correlation with the product of the aniline point and the specific gravity.

** The Watson characterisation factor is an approximate index of paraffinicity, with high volumes corresponding to high degrees of saturation. The characterisation factor is a satisfactory approach for correlating the physical and chemical properties of straight run petroleum fractions. It does not, however, accurately characterise fractions containing appreciable amounts of olefins, diolefins or cyclic compounds.

The 'true boiling point distribution' was determined by G.C. according to the method of Jackson *et al*. [5] who found excellent agreement between boiling point distribution by G.C. with 'true boiling point distribution' curves obtained by conventional distribution.

T A B L E   3

MAJOR HYDROCARBON COMPONENTS AND ELEMENTAL ANALYSIS OF B. BRAUNII OIL

Compound	Molecular Weight	% Mass	GC Retention Index
Isobotryococcene	466	4	2708
Botryococcene	466	9	2730
$C_{34}H_{58}$	466	11	2762
$C_{36}H_{62}$	494	34	2920
$C_{36}H_{62}$	494	4	–
$C_{37}H_{64}$	508	~ 20	2962
Other hydrocarbons		18	–

Element	% Mass
Carbon	86.38
Hydrogen	11.96
Oxygen	1.1
Nitrogen	0.17
Sulphur	< 0.1

T A B L E   4

TRUE BOILING POINT DISTRIBUTION BY G.C. OF UNCRACKED OIL

Recovered Point	(^{o}C)
5% Recovered Point	54
10% " "	77
20% " "	430
30% " "	447
40% " "	452
50% " "	456
60% " "	462
70% " "	466
80% " "	472
90% " "	476
95% " "	481
100% " "	527

Yield of I.B.Pt. -190.5^{o}C Fraction, Mass % = 17.0

Yield of 190.5^{o}C-232°C Fraction, Mass % = 0.2

Yield of 232°C-315.5^{o}C Fraction, Mass % = 0.6

Yield of > 315.5^{o}C Fraction, Mass % = 82.2

T A B L E   5

TESTING OF THE TOTAL SAMPLE

Method	Test	Result
ASTM D1298-67	S.G. (60/60)	0.7542
	API gravity	56.1

True boiling point by G.C.			(^{o}C)
	5%	Recovered point	30
	10%	" "	50
	20%	" "	84
	30%	" "	108
	40%	" "	128
	50%	" "	149
	60%	" "	172
	70%	" "	199
	80%	" "	228
	90%	" "	269
	95%	" "	298
	100%	" "	485

Yield of I. B. Point-191°C Fraction,   % Mass = 67.0

Yield of 191°C-232°C Fraction,   % Mass = 14.3

Yield of 232°C-315°C Fraction,   % Mass = 15.7

Yield of > 315°C Fraction,   % Mass = 3.0

T A B L E   6

ASTM TESTING OF THE GASOLINE CUT[+]

Method	Test	Result
ASTM D941-55	S.G. (60/60)	0.7364
	API gravity	60.7
ASTM D1319-70	Aromatics, % volume	1.62
	Olefins, % volume	< 0.2
	Micro D86 on 10 ml sample	(^{o}C)
ASTM D86-67 (modified)	Initial Boiling Point	44
	5% Recovered point	57
	10% " "	69
	20% " "	88
	30% " "	103
	40% " "	115
	50% " "	128
	60% " "	139
	70% " "	149
	80% " "	163
	90% " "	179
	Final Boiling Point	180
	% recovered	92.5
	% residue	4.5
	% loss	3.0

[+] Primary distillation details. Micro D86-67 on 20 ml of whole crude.
Cut point VT 20-191°C, % volume cut 0-62.5.

T A B L E   7

API TESTING OF THE GASOLINE FRACTION

Method	Test	Result
API Testing TECH. DATA BOOK FIGS. 2B1.1, 2B2.1	Calculation of characterising parameters on $20-191^{\circ}C$ fraction volumetric average boiling point, $(^{\circ}C)$	 126
	mean average boiling point, $(^{\circ}C)$	116
	C:H mass ratio	5.7:1
	aniline point, $(^{\circ}C)$	54
	molecular weight	112
	aniline gravity constant	7891
	Watson characterising factor	12.1

T A B L E   8

ASTM TESTING OF THE KEROSINE CUT[+]

Method	Test	Result
ASTM D941-55	S.G. (60/60) API gravity	0.8032 44.7
ASTM D1319-70	Aromatics, % volume Olefins, % volume	4.65 < 0.2

[+] Primary distillation details. Micro D86-67 on 20 ml of whole crude sample.
Cut point VT, 191-232°C; % volume cut 17.5, yield cut range (% vol.)
62.5-80.0%.

COMPOSITION OF THE GASOLINE FRACTION BY G.C.

Component	% Mass[+]	Component	% Mass[+]
Propane*	0.015	1-Cis-2-Cis-4-Trimethylcyclopentane[b]	("
Isobutane*	0.307	2,2,4-Trimethylhexane	0.383
n-Butane*	0.502	1,1-Dimethylcyclohexane[c]*	(1.611
Isopentane*	3.635	1-Trans-4-Dimethylcyclohexane[c]	("
n-Pentane*	1.768	1-Cis-3-Dimethylcyclohexane	1.068
2,2-Dimethylbutane*	0.320	1-Methyl-Trans-3-Ethylcyclopentane[d]*	(1.558
Cyclopentane[a]	(5.087	1-Methyl-Cis-3-Ethylcyclopentane[d]*	("
2,3-Dimethylbutane[a]*	("	1-Methyl-Trans-2-Ethylcyclopentane[d]	("
2-Methylpentane[a]*	("	1-Methyl-1-Ethylcyclopentane[d]	("
3-Methylpentane*	2.634	1-Cis-2-Cis-3-Trimethylcyclopentane*	1.012
n-Hexane*	3.011	n-Octane*	1.002
2,2-Dimethylpentane	0.287	1-Trans-2-Dimethylcyclohexane*	1.113
Methylcyclopentane*	1.549	1-Trans-3-Dimethylcyclohexane[e]*	(0.327
2,4-Dimethylpentane*	0.595	1-Cis-4-Dimethylcyclohexane[e]	("
Benzene	0.026	2,2-Dimethylheptane[f]	(0.516
3,3-Dimethylpentane*	0.200	2,4-Dimethylheptane[f]*	("
Cyclohexane*	0.992	3-Propylcyclopentane	0.242
2-Methylhexane*	2.897	2,2,3-Trimethylhexane	0.260
2,3-Dimethylpentane	1.250	2-Methyl-4-Ethylhexane	0.238

T A B L E 9

(Continued)

Component	% Mass[+]	Component	% Mass[+]
1,1-Dimethylcyclopentane	0.264	2,6-Dimethylheptane[g]	(0.667
3-Methylhexane*	3.437	4,4-Dimethylhexane[g]	("
1-Cis-3-Dimethylcyclopentane*	0.889	2,5-Dimethylheptane[h]	(2.050
1-Trans-2-Dimethylcyclopentane*	1.071	3,5-Dimethylheptane[h]	("
1-Trans-2-Dimethylcyclopentane*	0.845	1-Cis-2-Dimethylcyclohexane*	-
n-Heptane*	1,658	3,3-Dimethylheptane	0.357
1-Cis-2-Dimethylcyclopentane	0.217	n-Propylcyclopentane	-
2,2-Dimethylhexane	0.412	Ethylcyclohexane[i]	(0.931
1,1,3-Trimethylcyclopentane*	0.463	Ethylbenzene[i]	("
Methylcyclohexane*	1.445	1,1,4-Trimethylcyclohexane	0.379
2,5-Dimethylhexane*	-	2,3,4-Trimethylhexane	0.287
2,4-Dimethylhexane*	0.626	Methylcycloheptane	0.973
Ethylcyclopentane*	0.845	3,3,4-Trimethylhexane	0.980
2,2,3-Trimethylpentane	-	2,3-Dimethylheptane[j]	(0.020
1-Trans-2-Cis-4-Trimethylcyclopentane*	0.519	3,4-Dimethylheptane[j]	("
3,3-Dimethylhexane	0.863	p-Xylene	0.063
Toluene	0.265	m-Xylene	0.859
1-Trans-2-Cis-3-Trimethylcyclopentane*	0.519	4-Ethylheptane	-
1,1,2-Trimethylcyclopentane*	(0.011	4-Methyloctane	1.437

T A B L E   9

(Continued)

Component	% Mass[+]	Component	% Mass[+]
2,3,4-Trimethylpentane	(0.011	2-Methyloctane	0.930
2,3-Dimethylhexane*	0.654	3-Ethylheptane	0.294
2-Methyl-3-Ethylpentane	0.147	3-Methyloctane	1.232
2-Methylheptane*	1.874	n-Nonane	0.651
4-Methylheptane	0.776	Isopropylbenzene	0.132
3-Ethylhexane	0.348	n-Propylbenzene	0.021
3-Methylheptane*	2.401		
1-Cis-2-Trans-4-Trimethylcyclopentane[b]	0.550		
o-Xylene	0.279		
1-Trans-2-Cis-3-Trimethylcyclohexane	0.586		
1-Trans-2-Cis-4-Trimethylcyclohexane	0.925		
1,1,2-Trimethylcyclohexane	0.327		
		Unidentified Components Eluting before n-Nonane	7.242
		Unidentified Components Eluting between n-Nonane and n-Decane	17.126
		Unidentified Components Eluting after n-Decane	6.349

Each superscript (a-h) respectively represents a group of hydrocarbons unresolved by gas chromatography.

[+] The use of four significant figures has been retained for internal consistency of the table.

* Components confirmed by GC-MS.

T A B L E   10

PONA[+] COMPONENT CLASSIFICATION

Category	Volume %
Paraffins[++]	49.5
Olefins*	0.1
Naphthenes[++]	19.8
Aromatics*	1.4
Unidentified	29.2
Total	100.0

[+] PONA = Paraffin, Olefin, Naphthene and
Aromatic Compounds

[++] Based on Table 9.
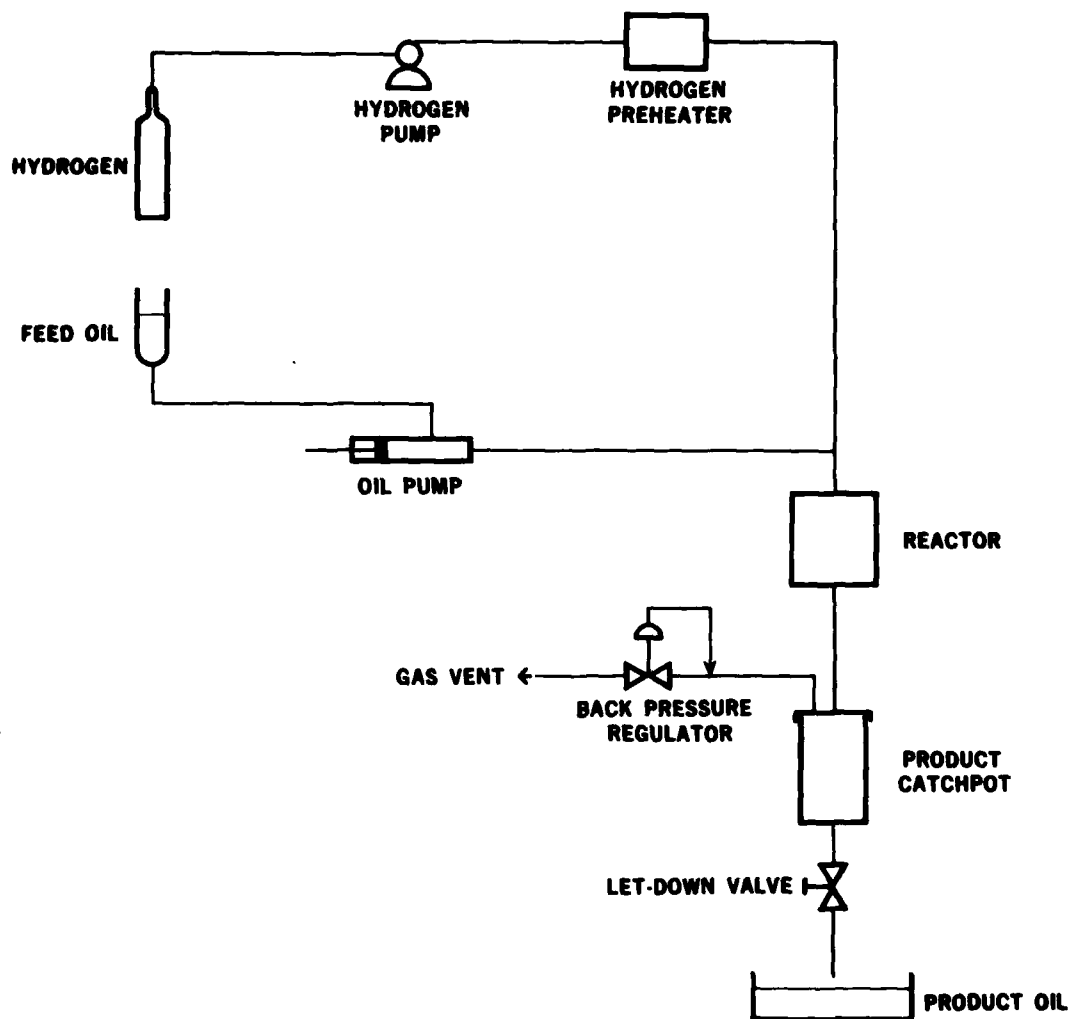
* Based on FIA Analysis

T A B L E   11

A COMPARISON OF SOME SYNTHETIC GASOLINES (REF. 4)

Gasoline Source	Olefins % y/y	Paraffins % y/y	Napthenes % y/y	Aromatics % y/y	RON Clear
Hydrogenated Brown Coal Tar	4.8	34.0	41.5	19.7	86
Hydrogenated Black Coal	0.3	23.0	54.0	22.7	–
Bass Strait Crude (straight-run)	0.0	45.4	40.7	13.9	54
Hydrocracked B. braunii oil[++]	< 0.2	68.5[+]	30.0[+]	1.4	82

[+] Partially estimated – see text.

[++] Results of present study

HYDROGEN

HYDROGEN
PUMP

HYDROGEN
PREHEATER

FEED OIL

OIL PUMP

REACTOR

GAS VENT ←

BACK PRESSURE
REGULATOR

PRODUCT
CATCHPOT

LET-DOWN VALVE

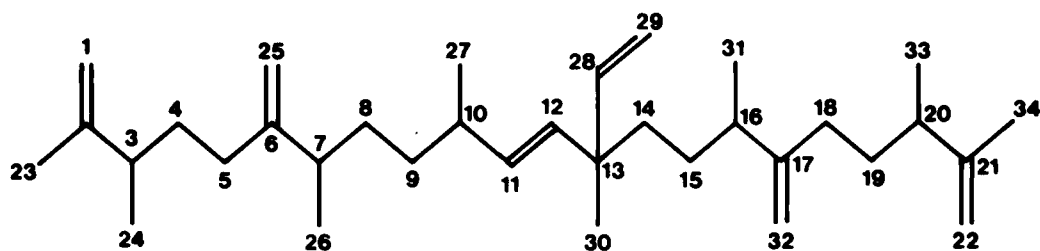PRODUCT OIL

Fig.1 Flowsheet of Pilot Hydro-Cracker

Fig.2   Structure of Botryococcene, $C_{34}H_{58}$
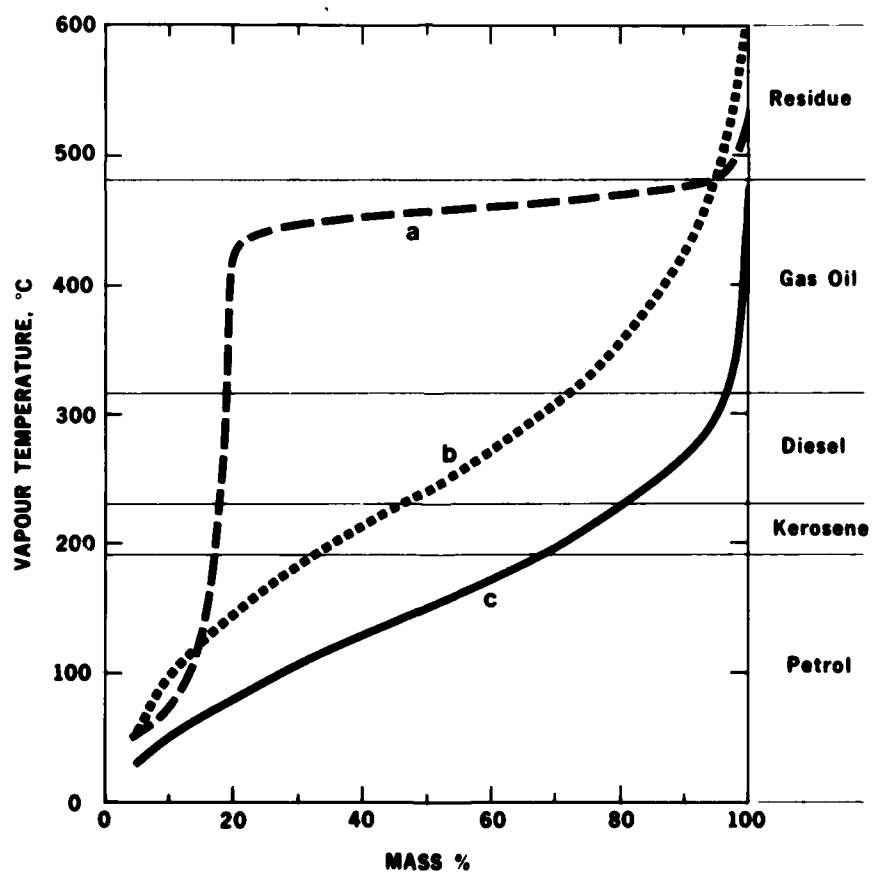

Fig.3   Comparison of Boiling Point Ranges of:-

a. Botryococcus Oil

b. Hydro-cracked Botryococcus Oil

c. Typical Bass Strait crude Oil

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